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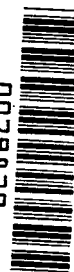


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DETERMINATION OF HEAT-SHIELD CHAR-FRONT RECESSION WITH A NUCLEONIC TECHNIQUE

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ABSTRACT

A discussion of the development of a radio-nuclide technique is presented, showing how to determine the amount of Apollo heat-shield virgin-material recession (char) occurring during an atmospheric re-entry. This technique utilizes a radioactive source inserted into the heat shield. This source decomposes and outgasses under the same conditions as does the heat-shield material. As portions of the source are removed through the processes of decomposition and sublimation, total radioactivity remaining will decrease at a rate proportional to the amount of virgin material remaining in the heat shield. A comprehensive evaluation program, using arc-jet, rocket-exhaust, and oxygen-acetylene torch facilities, is presented. Various sources of error are analyzed, and estimates of total system accuracy are developed.

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SUMMARY

A technique has been developed which allows continuous monitoring of the char-front progression through the Apollo heat-shield material during reentry. The performance of this measurement technique was tested under various simulated reentry conditions in both oxygen-acetylene and arc-jet facilities. The technique always defined the char front within a 0.06-inch accuracy, independent of the heating conditions.

The technique uses a radioactive source material which decomposes and sublimes under the same heating conditions as the heat-shield material. This radioactive material is encased in a small-diameter tube which is inserted in the heat shield perpendicular to the surface. As the source material decomposes and is removed, the total amount of radioactivity remaining at any time is proportional to the amount of virgin material remaining in the heat shield.

A specific definition of the actual parameter being measured and the relation of this parameter to the charring process of the heat-shield material are presented. Various sources of error relative to the nucleonic technique are analyzed, and estimates of total system accuracy are developed.

INTRODUCTION

As a result of the high velocities and associated extreme heating conditions experienced by the Apollo vehicle during reentry into the earth's atmosphere, from both earth-orbit and lunar trajectories, it has become increasingly important to extend the accuracy and resolution of the techniques used to measure the heat-shield performance. The parameters of prime interest are heat flux, surface recession, thermal gradients, pressure, and char-front recession. This report is confined to a discussion of the development of a measurement technique which utilizes a radionuclide to determine continuously the location of the char front during reentry. The ability of this technique to define the char-front location continuously will provide information concerning any small-scale fluctuations in the char-front recession. Also, the recession rate at any time can be measured directly, instead of being extrapolated from data points, as is required when a discrete type (step-output) system is used.

An experimental investigation was conducted to select a source material which could be radioactively tagged and which would, as closely as possible, exhibit the same thermal decomposition properties as the Apollo heat-shield material. The testing during this investigation was conducted in the Manned Spacecraft Center Arc-Jet Facility, by using inert-source substitutes which replace the radioactive isotope with an inert isotope of the same element. Further testing was conducted in additional high-temperature facilities by using both inert and radioactive sources.

This report describes the performance of the nucleonic char-measurement technique, the laboratory procedure for determining the accuracy of the system, and the disadvantages, as well as the advantages, inherent in this approach to the continuous measurement of char-front recession.

DEFINITION OF THE CHAR FRONT

To determine the location of the region generally known as the "char front" in the Apollo heat-shield material, a specific definition of this front is essential.

Three of the basic processes by which the heat shield protects the spacecraft are: (1) thermal insulation, (2) endothermic reactions of the constituents of the heat shield, and (3) mass loss. As these processes take place under reentry conditions, the physical characteristics of the virgin, or original, heat-shield material change. The endothermic reactions produce the following results: a loss of mass and, therefore, a decrease in density; a change in the color from relatively light to black; a loss in structural integrity; an internal temperature rise caused by degradation of the thermal insulation properties. The location in the heat-shield material where these changes occur was chosen as the definition of the char front for this study; however, a second char front does exist.

As more heat is forced into the heat shield, the endothermic reactions essentially cease. This cessation is accompanied by an increase in the hardness of the char and, therefore, an increase in structural integrity. Also, the electrical resistivity of the heat-shield material decreases sharply at this point. The material from this point to the surface of the heat shield consists of relatively inert compounds, which are vaporized or removed by dynamic pressures on the surface. The two char fronts and the decomposition zone between them are apparent, as is shown in figure 1.

Previously developed char-front measurement systems detect the rapid change in electrical resistivity of the second char front, which is defined by the interface between the decomposition zone and the inert char layer. These systems consist of several pairs of electrical leads positioned at various depths through the thickness of the heat shield. As the interface between the char layer and decomposition zone reaches each pair of leads, a resistance-sensitive circuit is energized. This technique can provide only step or event function data and cannot provide the more desirable continuous data.

Also, since the rapid decrease in resistivity occurs at the interface between the inert char layer and the decomposition zone, little information can be provided about the virgin material recession.

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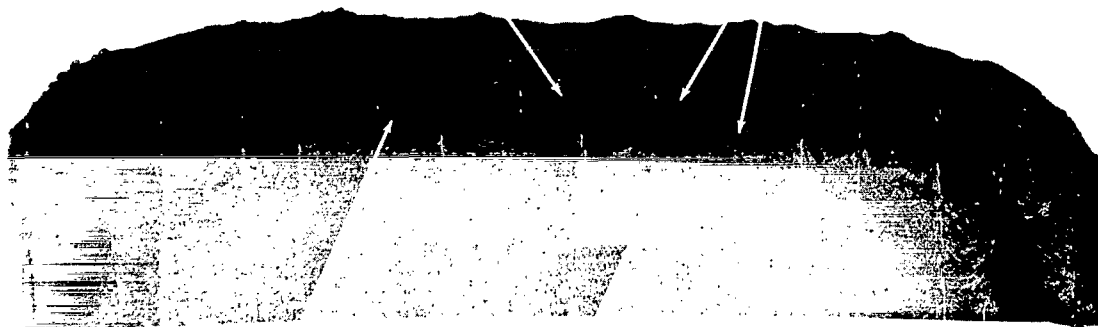


Figure 1. - Photograph of a sectioned heat-shield test model indicating visual difference between the char layer and the virgin material as well as the two char fronts.

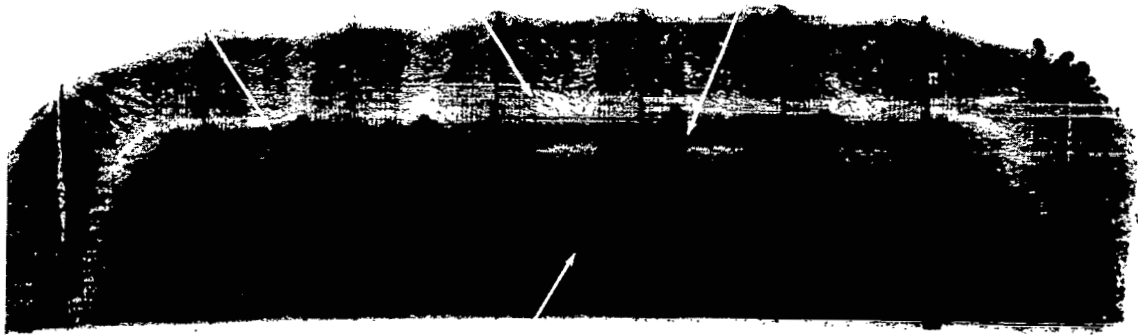
LABORATORY PROCEDURE USED TO LOCATE THE CHAR FRONT

Once the physical characteristics of the char front had been defined, methods of measuring these were devised, and one optimum technique was chosen to evaluate the performance of the nuclear char-front measurement technique.

After the arc-jet tests, changes in density, color, and hardness were used as a reference to determine the actual location of the char front. Although these three parameter changes occurred at the same location, the density-change measurement offers more information. The relative change in density between the char and the virgin material is very definite and can easily be detected by the use of X-ray inspection, as is shown in figure 2. The ratio of the density of the virgin material to the char material is approximately 2.5 to 1. Radiographic inspection functioned in two ways: (1) it facilitated inspection of the heat-shield model before the test, as shown in figure 2(a), and (2) it provided a precise method of measuring the amount of test source or inert source substitute remaining and its relation to the actual char-front



(a) X-ray radiograph before tests



(b) X-ray radiograph after test indicating the techniques used to locate the char front and the location of the char sensors

Figure 2. - Sectioned heat-shield model.

location, as shown in figure 2(b). This measurement procedure offered the distinct advantage of determining the amount of char-front recession, without disturbing the material at the point of interest, as well as of providing a permanent record of each test result. The radiographic inspection procedure was, therefore, chosen as the primary method for analyzing the test results, with the color and hardness variations used as supplementary information.

DEVELOPMENT TESTING AND RESULTS

Since the heat shield forms char by sublimation of its own materials, the selection of a char-measurement source had to be based on the testing of various materials which decompose, or sublime, within the generally accepted char temperature range of 600° to 1200° F. This development also required that the char-measurement source not only possess the proper thermal decomposition properties, but also that one or more compounds in it possess radionuclides with an acceptable nuclear decay scheme. Several types of small-diameter tubes were also tested which would facilitate preparation and handling of the source material.

As a result of the relative uncertainty of the heating conditions and of the dynamic pressures at any particular time during reentry, screening tests were conducted, using the criterion that failure of any source material to sublime or to decompose, when the heat-shield material charred under any heating condition would exclude that source material from further testing.

Arc-jet tests were conducted at the Manned Spacecraft Center, in which the various "candidate" sensor materials and tubes were compared. In each test, mercuric sulfide, mixed with a plastic binder contained in a 0.063-inch-outside-diameter teflon tube, was found to define the char front to within 0.02 inch. Radiographic inspection then indicated that all of the mercuric sulfide in the char layer between the char front and the ablation surface had been lost, and that the source material in the virgin material was intact. In addition, an emission spectrographic analysis of the char layer that surrounds the source configuration was unable to detect any mercury.

Mercuric sulfide combined with the plastic binder in a teflon tube offered several distinct advantages. The mercuric sulfide sublimates at approximately 1078° F (ref. 1) and outgasses through the char layer. A possibility existed that the mercuric sulfide would be chemically reduced to metallic mercury in the char layer. However, the metallic mercury has a boiling point which is about 370° F lower than the sublimation temperature of mercuric sulfide; therefore, no mercury would remain trapped in the char layer. Mercuric sulfide can be radioactively tagged with mercury 203. This radionuclide emits gamma photons of an energy which can be monitored with either scintillation or Geiger-Mueller detectors. Another advantage of this configuration is that, as it decomposes, the plastic binder forms a carbon residue similar in physical form to the heat-shield char. This carbon residue prevents abnormal heat-shield performance in the area surrounding the source material.

A test program was conducted to determine the accuracy with which the selected char sensor would define the heat-shield char front under the environments encountered during reentry. Since presently available arc-jet and rocket-exhaust facilities can only simulate, at best, a portion of the true reentry environment at one time, the heating rate, enthalpy, shear, and temperature must be obtained individually in separate tests. This separate testing precludes a practical experimental determination of the combined effect of these parameters on any heat-shield instrumentation.

The initial phase of the test program consisted of testing the final source configuration with an inert-source substitute in various arc-jet and rocket-exhaust facilities. A range of reentry conditions of enthalpy, heating rate, surface temperature, shear force, and ambient pressure was obtained during these tests. A total of twenty-two inert-source substitutes was tested in this phase. Post-test X-ray inspection of the heat-shield models indicated a maximum error of 0.03 inch between the end of the inert source and the position of the char front, with an average error of 0.01 inch and a root-mean-square (rms) error of 0.02 inch. This measurement error was found to be independent of the source length. Typical results of these tests and the conditions of each test are shown in table I.

TABLE I.- TYPICAL ARC-JET AND ROCKET EXHAUST TEST CONDITIONS
AND RESULTS FOR INERT SOURCES

Heating rate, Btu/ft ² -sec	Enthalpy, Btu/lb	Shear, psi	Run time, sec	Surface reces- sion, in.	Actual char re- cession, in.	Indicated char re- cession, in.	Char meas- urement error, in.
60	3 530	0.75	90	0.30	0.44	0.42	0.02
200	5 000	.00	50	.17	.42	.45	.03
250	6 000	.00	80	.20	.40	.40	.00
280	7 000	.00	75	.23	.43	.42	.01
298	20 700	.50	170	.55	.72	.70	.02
355	9 500	.00	38	.23	.35	.37	.02
375	3 510	5.25	36	.55	.60	.60	.00
375	3 510	5.25	36	.60	.67	.67	.00
475	9 200	.00	30	.22	.35	.35	.00
545	12 758	.00	30	.23	.35	.35	.00
561	25 500	.50	170	.50	.75	.75	.00
570	24 600	.50	170	.65	.82	.82	.00

An important result of these tests was that no surface irregularities which would jeopardize the heat-shield performance resulted from the introduction of the inert sources. This was true even in the high shear-force tests.

A second phase of testing involved the fabrication of radioactive sources and the testing of these sources in heat-shield material in a char-producing environment. These tests were necessary, not only to verify that the char-front position can be determined by the nucleonic approach, but also to determine the total accuracy of a nuclear char-measurement system, which would consist of a radioactive source, a gamma-sensitive detector, and a rate-meter readout. Since testing radioactive sources in arc-jet facilities was impractical, an oxygen-acetylene torch enclosed in an exhaust hood fitted with absolute filters was used. A sketch of the test setup is shown in figure 3.

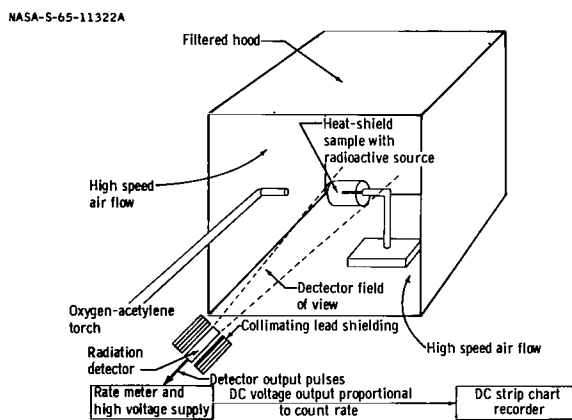


Figure 3. - Sketch of test setup for testing the radioactive char measurement technique.

system were also inherent in the detector and readout system used in these tests.

The average total system error resulting from these tests was 0.02 inch, with an rms error of 0.03 inch and a maximum error of 0.06 inch. The test results and testing conditions are shown in table II. Typical output data for various heating rates are shown in figure 4.

A test was conducted to determine what effect the radiation from the radioactive mercury would have on the teflon tube and plastic binder. Twelve inert char-source configurations were exposed to gamma radiation in a 100-curie, cobalt 60 irradiator. The air equivalent dose was varied from 0 to 1 500 000 roentgens. The testing of these sources in the Apollo material indicated that no detectable effect in the performance of the source resulted from the exposure to gamma radiation.

TABLE II.- TEST DATA AND RESULTS FOR THE NUCLEAR CHAR-FRONT MEASUREMENT SYSTEM

Heating rate, Btu/ft ² -sec	Run time, sec	Actual char recession, in.	Indicated char recession, in.	Char measurement error, in. (a)
25	90	0.13	0.12	-0.01
25	190	.20	.20	.00
85	120	.35	.38	+.03
85	240	.55	.57	+.02
85	240	.45	.44	-.01
110	120	.55	.58	+.03
110	180	.82	.86	+.04
110	180	.62	.61	-.01
140	90	.30	.32	+.02
190	90	.50	.53	+.03
190	120	.60	.64	+.04
190	120	.55	.53	-.02
210	60	.40	.43	+.03
210	90	.50	.52	+.02
210	120	.60	.54	-.06
215	150	.35	.40	+.05
275	60	.33	.33	.00

(a) + indicates that actual char-front recession was more than the system-indicated recession.

- indicates that actual char-front recession was less than the system-indicated recession.

ERROR ANALYSIS

Two basic sources of error are inherent in a continuous char-front measurement system which utilizes nuclear counting techniques. These errors are: (1) the error between the front end of the source configuration and the position of the char front, and (2) the error introduced by the statistical

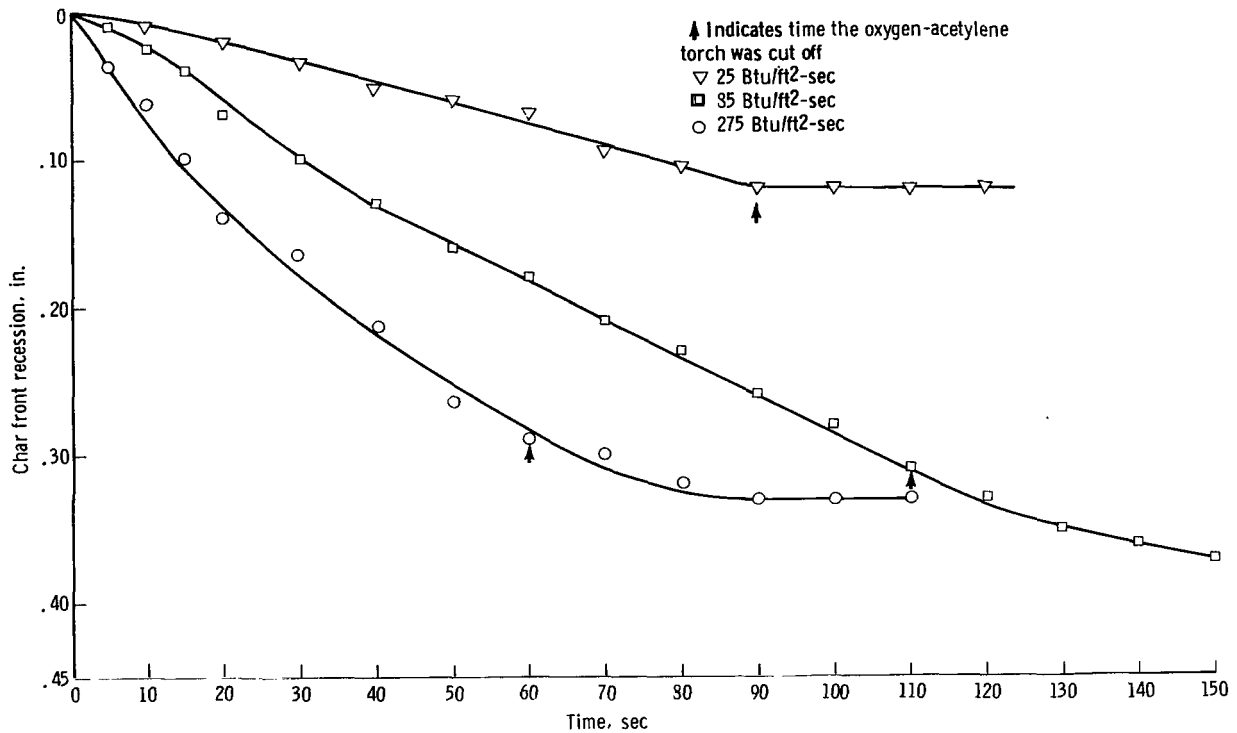


Figure 4. - Experimental data.

nature of a nuclear count-rate system. In order to determine the maximum accuracy which can be expected from the nuclear char-front measurement system, the two sources of error must be analyzed individually, and then combined. The resulting total error can then be compared to the results from the testing of the radioactive sources.

The first source of error can be determined only experimentally. Data from a portion of the test program, and consisting of the arc-jet and rocket-exhaust tests of 22 inert-source substitutes, provide this information. The experimentally determined rms error $\sigma_{a(\text{exp})}$ between the front end of the source configuration and the actual char-front position was found to be approximately 0.02 inch.

The second source of error can be determined theoretically from the parameters of the detector and of the rate-meter readout system. The test equipment used to monitor the radioactive sources operated at a full-scale count rate of 5000 counts per second, and with a time constant of 1.0 second.

The rms counting error $\sigma_{c(\text{theor})}$ of the test equipment is given by the following equation (refs. 2 and 3):

$$\sigma_{c(\text{theor})} = \sqrt{\frac{r}{2 RC}}$$

where r is the count rate of the rate meter in counts per second, and RC is the time constant of the rate meter in seconds. Therefore, for a full-scale count rate of $r = 5000$ counts per second, with a time constant of $RC = 1.0$ seconds,

$$\sigma_{c(\text{theor})} = \sqrt{\frac{5000}{2(1)}} = \sqrt{2500} = 50 \text{ counts per second}$$

The percent rms counting error is then $\frac{50}{5000} \times 100$ percent or 1.0 percent, that is, 0.01 inch for a 1-inch source length.

Table III shows that as the source is lost and as the count rate of the rate meter decreases, the percent rms error increases. However, since the length of the source also is decreasing, the actual measurement error in inches remains relatively constant.

TABLE III.- ROOT-MEAN-SQUARE COUNT ERROR AS A FUNCTION OF
COUNT RATE AND OF SOURCE LENGTH

Source length, in.	Count rate, counts/sec	Rms counting error	
		Percent	in.
1.00	5000	1.00	0.010
.75	3750	1.15	.009
.50	2500	1.41	.007
.25	1250	2.00	.005

These two sources of error $\sigma_{a(\text{exp})}$ and $\sigma_{c(\text{theor})}$ can be combined to give a total minimum rms error σ_T as can be deduced from the measurement technique applied. Using the rms counting error $\sigma_{c(\text{theor})} = 0.01$ inch between the front end of the source and the actual char-front location, the total minimum rms error to be expected is given by the following equation (ref. 2):

$$\sigma_T = \sqrt{\sigma_{a(\text{exp})}^2 + \sigma_{c(\text{theor})}^2}$$

or

$$\sigma_T = \sqrt{(0.02)^2 + (0.01)^2} = 0.022 \text{ inch}$$

Results from the seventeen radioactive source tests shown in table II indicate rms error of 0.03 inch. Therefore, the actual measurement error approaches the theoretical accuracy limit.

However, other sources of error, such as detector non-linearity, non-uniform source-material distribution, and source-detector geometry, do exist. But such errors are minor, and can be corrected by proper calibration procedures. Any non-uniformity of the radioactive material through the length of the source can be eliminated by careful preparation. Sources were prepared with linearities varying by less than 2 percent along the total length. Methods for the detector calibration and for the geometry calibration have been developed, and are relatively straight forward.

DISCUSSION OF RESULTS

This development program has produced a technique for the determination of the Apollo heat-shield char front recession. Although no method was available to monitor continuously the actual char front location during a test, the char front progression data as determined by the developed nuclear measurement technique were a reasonable indication of the true heat-shield performance. The curve for the higher heating rate of 275 Btu/ft²-sec in figure 4 shows a rapid char front recession until a protective char layer was formed. As the heating rapidly formed the char, the char front progression rate decreased. The two lower heating rate curves shown in figure 4 did not exhibit as rapid a char progression at first, but more closely approached a continuous straight line. The slopes of these lines are directly proportional to the heating rates. The tests also verified that the amount of activity remaining in the source was an accurate indication of the amount of virgin material remaining at any time.

Several significant results of the testing of the developed nuclear approach to char front measurement should be pointed out. Over the relatively large range of heating rates, enthalpies, shear forces, testing durations, and the various types of sources of heating utilized in the test program, no irregularities in the performance of the Apollo heat-shield material resulted from the presence of this source configuration. This is important since abnormal heat-shield performance caused by any heat-shield measurement instrumentation must preclude the use of such instrumentation since proper operation of the heat shield is critical to the success of all Apollo missions. Another important result was that over the range of testing conditions, the nuclear char front measurement technique did not appear to be restricted to a limited range of any of the test conditions, such as

heating rate. This technique defined the char-front interface between the decomposition zone and the virgin material under all test conditions.

In a parallel effort the National Aeronautics and Space Administration has developed a nucleonic technique for the measurement of heat-shield surface recession (ref. 4). The utilization of these two developed techniques could provide continuous information on char-front recession, ablation-surface recession, and char-layer thickness.

CONCLUDING REMARKS

There are several advantages to this nucleonic technique for measurements of char-front progression. No holes in the substructure of the Apollo vehicle are required - as would be necessary for instrumentation utilizing electrical leads. Probably the most significant advantage of this technique is the fact that it is a continuous type of measurement, whereas other char-measuring systems are step or event function measurements based on detection of the movement of the char-front interface at several predetermined points through the thickness of the heat shield.

Manned Spacecraft Center
National Aeronautics and Space Administration
Houston, Texas, November 4, 1965

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